



IN THE U.S. PATENT AND TRADEMARK OFFICE

Applicants : YAMADA et al.

Serial No. : 10/514,411

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For : PHOTSENSITIVE RESIN COMPOSITION FOR FORMING  
A LASER ENGRAVABLE PRINTING ELEMENT

Art Unit : 1752

Examiner : Connie P. Johnson

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DECLARATION UNDER 37 C.F.R. 1.132

The undersigned, Hiroshi Yamada, a Japanese citizen residing at 2-9-13 Koubaracho, Mishima-shi, Shizuoka-ken, Japan, declares and say:

I was graduated in March 1989 from the Doctor course of Tokyo Institute of Technology.

I entered Asahi Kasei Kabushiki Kaisha in April 1989. I have been engaged in research and development of resin compositions.

I am well familiar with the present case.

I read and understood the Office Action dated June 14, 2007 and references cited therein.

I have made observations on the properties of various inorganic particles to demonstrate that a number average particle diameter, an average pore diameter and a pore volume of inorganic particles are independent properties without any correlation among them and that the pore diameter and pore volume of particles cannot be contemplated from the number average particle diameter of the particles. The materials, method and results are described in a paper attached hereto and marked "Exhibit 1".

I have also made observations on the criticality of using inorganic porous particles having a specific number average particle diameter, average pore diameter and pore volume for suppressing the generation of liquid debris during the laser engraving of a printing element and lowering the amount of surface tack, with reference to Example 1 and Comparative Examples 4 and 5 of the present specification. The method and results are described in a paper attached hereto and marked "Exhibit 2".

From the results of Exhibits 1 and 2, it can be fairly concluded:

(1) that a number average particle diameter, an aver-

age pore diameter and a pore volume are independent properties without any correlation among them (Exhibit 2);

(2) that, with respect to inorganic porous particles, the average pore diameter and the pore volume are independent properties without any correlation therebetween (Exhibit 1);

(3) that inorganic particles having a number average particle diameter of not more than 10  $\mu\text{m}$  do not necessarily have an average pore diameter of from 1 nm to 1,000 nm and a pore volume of from 0.1 ml/g to 10 ml/g (Exhibit 1);

(4) that the average pore diameter and the pore volume of inorganic particles cannot be contemplated from the number average particle diameter thereof (Exhibit 1);

(5) that the use of inorganic porous particles which simultaneously have an average pore diameter of from 1 nm to 1,000 nm, a pore volume of from 0.1 ml/g to 10 ml/g and a number average particle diameter of not more than 10  $\mu\text{m}$  is critical for suppressing the generation of liquid debris during the laser engraving of a printing plate and lowering the amount of surface tack (Exhibit 2).

The undersigned petitioner declares that all statements made herein of his own knowledge are true and that all statements made on information and belief are believed

to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Date: \_\_\_\_\_

\_\_\_\_\_  
Hiroshi Yamada



Exhibit 1

Observations on the properties of various inorganic particles to demonstrate that a number average particle diameter, an average pore diameter and a pore volume of inorganic particles are independent properties without any correlation among them and that the pore diameter and pore volume of particles cannot be contemplated from the number average particle diameter thereof

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1. Object of the observation:

The inorganic porous particles used in the present invention have an average pore diameter of from 1 nm to 1,000 nm, a pore volume of from 0.1 ml/g to 10 ml/g and a number average particle diameter of not more than 10  $\mu$ m.

The following observations are made to demonstrate that a number average particle diameter, an average pore diameter and a pore volume of inorganic particles are independent properties without any correlation among them and that the pore diameter and pore volume of particles cannot be contemplated from the number average particle diameter thereof.

## 2. Observations:

Each of commercially available products (which are inorganic particles) shown in Table A below was analyzed to determine an average pore diameter, a pore volume and a specific surface area thereof. Specifically, 2 g of the product as a sample was placed in a test tube and vacuum-dried for 12 hours by a pretreatment apparatus at 150 °C under 1.3 Pa or less. The pore volume, average pore diameter and specific surface area of the dried product were measured by "Autosorb-3MP" (manufactured and sold by Quantachrome Instruments, U.S.A.), wherein nitrogen gas was adsorbed on the product in an atmosphere cooled by liquid nitrogen. The specific surface area was calculated by the BET formula. With respect to the pore volume and average pore diameter, a cylindrical model was postulated from the adsorption isotherm during the elution of nitrogen. The pore volume and average pore diameter were calculated by the BJH method.

The results of the analysis are shown in the following Table A, together with number average particle diameter and oil absorption value which are described in the manufacturer's catalog. In Table A, the values which do not fall within the ranges defined in the present invention are shown with underlining.

Table A

Item	Tradename of inorganic particles	Number average particle diameter (nm)	Average pore diameter (nm)	Pore volume (ml/g)	Specific surface area (m <sup>2</sup> /g)	Oil absorption value (ml/100 g)
[1]	SOC-NPS-6MG(50) <sup>*3</sup>	0.1	1.6	2.0	1000	not determined
[2]	M.S.GEL EP-DF-3-1000AW <sup>*2</sup>	2.4	113	0.6	22	160
[3]	SYLYSIA 710 <sup>*1</sup>	2.8	2.5	0.44	700	100
[4]	Silton AMT25 <sup>*4</sup>	2.9	none	0.006	2.3	40
[5]	SUNSPHERE H31 <sup>*2</sup>	3.0	5	1.0	800	150
[6]	SUNSPHERE H33 <sup>*2</sup>	3.0	30	2.0	700	400
[7]	M.S.GEL EP-DF-3-2000AW <sup>*2</sup>	3.0	200	0.7	14	190
[8]	M.S.GEL EP-DF-3-1500AW <sup>*2</sup>	3.2	154	0.7	18	180
[9]	SYLYSIA 550 <sup>*1</sup>	3.9	7	0.8	500	160
[10]	SYLOSPHERE C-1504 <sup>*1</sup>	4.5	12	1.5	520	290
[11]	Silton JC50 <sup>*4</sup>	5.0	none	0.02	6.7	45
[12]	SYLYSIA 250USA <sup>*1</sup>	5.7	24	1.8	300	310

\*1: manufactured and sold by FUJI SILYSIA CHEMICAL LTD.

\*2: manufactured and sold by AGC Si-Tech. Co., Ltd.

\*3: manufactured and sold by SUMITOMO OSAKA CEMENT Co., Ltd.

\*4: manufactured and sold by Mizusawa Industrial Chemicals, Ltd.

As apparent from Table A above, the relationship between the number average particle diameter and each of the average pore diameter, pore volume, specific surface area and oil absorption value varies among different commercially available products which are inorganic particles. For example, items [3] to [7] (namely SYLYSIA 710, Siltan AMT25, SUNSPHERE H31, SUNSPHERE H33 and M.S.GEL EP-DF-3-2000AW) all have the same or similar number average particle diameter (2.8-3.0 mm), but none of the products have similar pore structure properties. Especially item [4] (Siltan AMT25) is a non-porous material having an average pore diameter below the detection limit and very low pore volume and specific surface area. Similarly, items [10] and [11] (namely SYLOSPHERE C-1504 and Siltan JC50) have similar number average particle diameter (4.5 mm and 5.0 mm, respectively), but item [10] is porous particles, whereas item [11] is non-porous particles having an average pore diameter below the detection limit and very low pore volume and specific surface area.

With respect to the inorganic porous particles, the relationship between the average pore diameter and the pore volume varies among different commercially available products. For example, the pore volume of item [6] (SUNSPHERE H33) is 2.0 ml/g, and this value is twice that of



item [5] (SUNSPHERE H31) which is 1.0 ml/g. Similarly, the average pore volume of item [6] (30 nm) is 6 times larger than that of item [5] which is 5 nm. Accordingly, the comparison between items [5] and [6] show that the average pore diameter increases with the increase in the pore volume. On the contrary, the comparison between items [8] and [9] (namely M.S.GEL EP-DF-3-1500AW and SYLY-SIA 550) show that the average pore diameter decreases with the increase in the pore volume. Specifically, the pore volume of item [9] (0.8 ml/g) is larger than that of item [8] (0.7 ml/g), but the average pore diameter of item [9] (7 nm) is much smaller than that of item [8] (154 nm).

### 3. Conclusion

From item 2. Observation above, it is apparent that a number average particle diameter, an average pore diameter and a pore volume are independent properties without any correlation among them. With respect to inorganic porous particles, the average pore diameter and the pore volume are independent properties without any correlation therebetween. In other words, inorganic particles having a number average particle diameter of not more than 10  $\mu\text{m}$  do not necessarily have an average pore diameter of from 1 nm to 1,000 nm and a pore volume of from 0.1 ml/g to 10 ml/g.

Accordingly, the average pore diameter and the pore volume of inorganic particles cannot be contemplated from the number average particle diameter thereof or vice versa.



Exhibit 2

Observations on the criticality of using inorganic porous particles having a specific number average particle diameter, average pore diameter and pore volume for suppressing the generation of liquid debris during the laser engraving of a printing element and lowering the amount of surface tack, with reference to Example 1 and Comparative Examples 4 and 5 of the present specification.

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1. Object of the observation:

The printing element of the present invention is made from a photocurable resin composition containing inorganic porous particles which simultaneously have an average pore diameter of from 1 nm to 1,000 nm, a pore volume of from 0.1 ml/g to 10 ml/g and a number average particle diameter of not more than 10  $\mu$ m.

The following observations are made to demonstrate the criticality of using such specific inorganic porous particles for suppressing the generation of liquid debris during the laser engraving of a printing element and low-

ering the amount of surface tack

## 2. Observations:

Laser engraved printing plates containing inorganic porous or nonporous particles were produced in Example 1 and Comparative Examples 4 and 5 of the present specification. Specifically, in Example 1 of the present specification, a printing element was produced using a porous microparticulate silica product (trade name: SYLOSPHERE C-1504) as the inorganic porous particles (c), and the obtained printing element was subjected to laser engraving for forming a relief pattern, thereby obtaining a printing plate. On the other hand, in each of Comparative Examples 4 and 5, a printing plate was produced in substantially the same manner as in Example 1 except that substantially nonporous particles were used instead of the inorganic porous particle (c).

The properties of the inorganic porous particles used in Example 1 and the inorganic nonporous particles used in Comparative Examples 4 and 5 are summarized in the following Table B, together with the properties defined in claim 1 of the present application.

**Table B**

	Inorganic porous material			
	Name (trade name)	Average pore diameter	Pore volume	Average particle diameter
Claim 1	---	1 to 1000 nm	0.1 to 10 ml/g	not more than 10 $\mu$ m
Example 1	Silica product (Sylosphere C-1504)	12 nm	1.5 ml/g	4.5 $\mu$ m
Comparative Example 4	Aluminosilicate (Silton AMT25)	---	0.006 ml/g	2.9 $\mu$ m
Comparative Example 5	Sodium calcium aluminosilicate (Silton JC50)	---	0.02 ml/g	5.0 $\mu$ m

Each of the produced printing plates was evaluated by the methods described at page 96, line 12 to page 99, line 2 of the present specification. The results of the evaluation are summarized in the following Table C.

**Table C**

	Frequency of wiping needed to remove the debris (BEMCOT impregnated with ethanol)	Tack on the relief printing plate after wiping (N/m)
Example 1	$\leq 3$	55
Comparative Example 4	10 <	350
Comparative Example 5	10 <	280

As can be seen from Table C above, when a printing plate was made from a printing element containing inorganic porous particles having an average pore diameter, a pore volume and a number average particle diameter within the specific ranges defined in claim 1 of the present specification (Example 1), the frequency of wiping needed to remove the engraving debris was not more than 3 times and the tack on the printing plate after wiping was as small as 55 N/m. On the other hand, when a printing plate was made from a printing element containing substantially non-porous inorganic particles (Comparative Examples 4 and 5), even after wiping the printing plate more than 10 times to remove the engraving debris, the tack on the printing plate was 280 N/m or more.

### 3. Conclusion

From item 2. Observation above, it is apparent that the use of inorganic porous particles which simultaneously have an average pore diameter of from 1 nm to 1,000 nm, a pore volume of from 0.1 ml/g to 10 ml/g and a number average particle diameter of not more than 10  $\mu$ m is critical for suppressing the generation of liquid debris during the laser engraving of a printing plate and lowering the amount of surface tack.